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IS 14721 (1999): Zinc Oxide for Ceramic Industry [CHD 9: Ceramicware]

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Indian Standard

ZINC OXIDE FOR CERAMIC INDUSTRY — SPECIFICATION

ICS 71.060.20; 81.060

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 1999 **Price Group 5**

Ceramicware Sectional Committee, CHD 9

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

Zinc oxide (ZnO) is naturally occurring zincite and is widely used in ceramic and its related industries as glazing material. It provides brightness and gloss to the ceramicware.

This standard has been prepared with a view to provide guidance to the ceramicware manufacturers for procurement of zinc oxide of proper quality as a suitable raw material. This standard may also serve as a guide to producers of such raw materials to cater to the needs of ceramic industry.

The composition of the technical committee responsible for the formulation of this standard is given in Annex J.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ZINC OXIDE FOR CERAMIC INDUSTRY — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for zinc oxide for ceramic industry.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
323 : 1959	Rectified spirit (*revised*)
1070 : 1992	Reagent grade water (*third revision*)
1574 : 1980	Glass weighing bottles (*first revision*)
2500 (Part 1) : 1992	Sampling and inspection procedures: Part 1 Attribute sampling plans indexed by acceptable quality level (AQL) for lot-by-lot inspection (*second revision*)
2781 : 1975	Glossary of terms relating to ceramicware (*first revision*)
4161 : 1967	Nessler cylinders
6128 : 1971	Desiccators
9755 : 1989	High density polyethylene (HDPE) woven sacks for packing fertilizers (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 2781 shall apply.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of white dry powder.

4.2 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the material shall be suitably packed in high density polyethylene (HDPE) woven sacks (*see* IS 9755).

5.2 Marking

The packages shall be marked with the following:

a) Name and type of the material;

b) Name of the manufacturer or his recognized trade-mark, if any;

c) Net mass of the material, in kg;

d) Month and year of manufacture; and

e) Batch number or lot number in code or otherwise.

5.2.1 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Lot

All the bags of the material blended at a time shall constitute a lot.

6.2 The scale of sampling and criteria for conformity of a lot shall be according to IS 2500 (Part 1).

6.2.1 All the characteristics given in this standard shall be tested on the composite sample.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in testing.

NOTE —'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Zinc Oxide for Ceramic Industry
(*Clause* 4.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Annex)
(1)	(2)	(3)	(4)
i)	Moisture content at 105 ± 2°C, percent by mass, *Max*	0.5	A
ii)	Residue on 45-micron IS sieve, percent by mass, *Max*	0.15	B
iii)	Powder density at 27°C	5.5 to 5.65	C
iv)	Loss on ignition at 850 ± 20°C, percent by mass, *Max*	0.3	D
v)	Zinc oxide (as ZnO), percent by mass, *Min*	99.5	E
vi)	Acidity (as H_2SO_4), percent by mass, *Max*	0.02	F
vii)	Iron (as Fe), percent by mass, *Max*	0.1	G
viii)	Metallic zinc	To pass the test	H

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MOISTURE CONTENT

A-1 APPARATUS

A-1.1 Weighing Bottle

Squat form, wide mouthed with ground glass stopper, diameter 70 mm (*see* IS 1574).

A-1.2 Oven

Capable of being maintained at 105 ± 2°C.

A-1.3 Balance

Accurate to one milligram or better.

A-1.4 Desiccator

Containing an efficient desiccant, such as silica gel or dehydrated calcium chloride (*see* IS 6128).

A-2 PROCEDURE

Heat the weighing bottle, with the stopper removed, in the oven at 105°C for two hours. Allow to cool in the desiccator, insert the stopper and weigh to the nearest one milligram. Spread 5 ± 1 g of the material in a uniform layer on the bottom of the weighing bottle, insert the stopper and weigh to the nearest milligram. Heat the weighing bottle, with the stopper removed, in the oven at 105 ± 2°C for two hours. Allow to cool in the desiccator, insert the stopper and weigh to the nearest milligram. Repeat heating and weighing until constant mass is obtained.

A-3 CALCULATION

$$\text{Moisture content at } 105^\circ\text{C, percent by mass} = \frac{100\,(m_1 - m_2)}{m_1}$$

where

m_1 = mass, in g, of the sample taken for test; and
m_2 = mass, in g, of the sample after heating.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF RESIDUE ON 45-MICRON IS SIEVE

B-1 OUTLINE OF THE METHOD

Measure the fineness of the material by sieving a known quantity of it through a 45-micron IS sieve.

B-2 APPARATUS

B-2.1 Gallie Porritt Sieve

The apparatus, as shown in Fig. 1, consists of a metal funnel terminating at the foot in a short cylindrical outlet, in which is inserted a shallow removable cup. To the bottom of the cup, a wire gauze of 45-micron of 25 mm diameter is soldered. Water under pressure of 150 to 350 kN/m^2 (approximately 1.5 to 3.5 kgf/cm^2) is supplied by a tube fitted with a nozzle designed to discharge a spreading jet throughout the wire gauze, and the tube is so arranged that the distance of the orifice in relation to the wire gauze may be adjusted. The tube is provided with a filter to ensure removal of any solid particles from the water. A second tube which is also provided with a filter is improvised to supply a gentle stream of water for wetting the powder and for supplying more water to the funnel during the progress of the test.

B-2.2 The area of the wire gauze shall be sufficiently large to prevent undue loss of pressure head.

B-3 PROCEDURE

Weigh accurately about 50 g ± 1 g of the material. Half fill the funnel with water from the subsidiary supply, then bring the central jet (*see* Fig. 1) into operation. Transfer the weighed material to the metal funnel either dry or in the form of a slurry with a suitable wetting agent. Turn off the subsidiary supply and, if necessary, adjust the height of the central jet so that the level of water in the funnel commences to fall when the level has fallen within 50 mm of the sieve, turn on subsidiary supply until the level is within 50 mm from the top of the funnel and maintain there until the liquid appears clear and then for a further period of one minute. Turn off the subsidiary supply and allow the level to fall again. When the funnel is empty, turn off the main jet and allow to drain. Remove the jet and examine for any large soft lump. If present, break up gently with a finger, replace the cup and wash with water from the subsidiary supply for two minutes. Transfer the grit on the sieve to a watch glass, dry at 105 ± 2°C and weigh.

B-4 CALCULATION

Residue on 45-micron sieve, percent by mass $= 100 \times \frac{m_1}{m_2}$

where

m_1 = mass, in g, of the residue left on the sieve, and

m_2 = mass, in g, of the material taken for test.

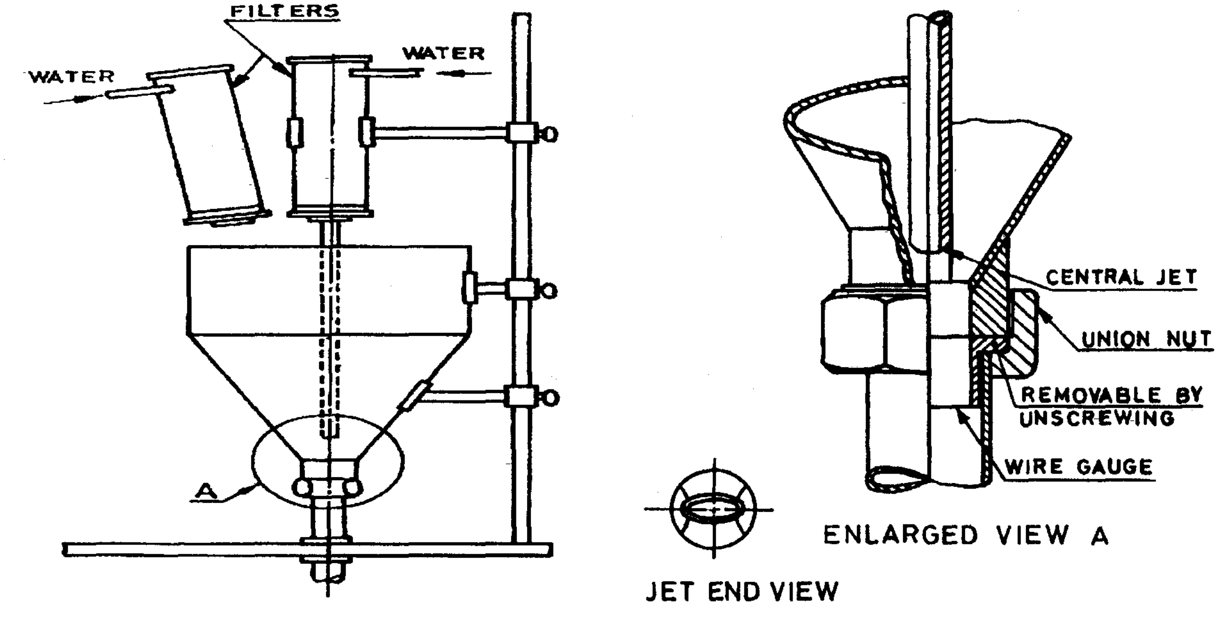

FIG. 1 GALLIE PORRITT SIEVE

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF POWDER DENSITY

C-1 APPARATUS

C-1.1 Analytical Balance

C-1.2 Pykonometer

50 ml capacity, with a ground capillary stopper.

C-1.3 Vacuum Source

To produce a vacuum of 12 to 25 mm of mercury.

C-2 PROCEDURE

C-2.1 Dry the pyknometer at room temperature after rinsing it with acetone or any quick drying solvent and then with hot air. Weigh the pyknometer and record the mass as m. Fill it with distilled water and note the water temperature, t. Insert the stopper and wipe the excess water from the tip in such a manner as not to withdraw any water from the capillary. Thoroughly dry any water adhering to the outside of the pyknometer and weigh. Record the mass as m_1.

C-2.2 Empty the pyknometer and dry it again. Introduce about 10 g of the dried sample and weigh with the stopper in position, recording the mass as m_2. Remove the stopper, fill the pyknometer with distilled water to one-fourth to one-half of its capacity, keep the pyknometer in a vacuum desiccator and apply vacuum to remove the entrapped air completely. Release the vacuum and take out the pyknometer from the desiccator and cool it to room temperature.

C-2.3 Fill the pyknometer with water. Insert the stopper, wipe the excess water from the tip and the outside of the pyknometer as above and weigh, recording the mass as m_3.

C-3 CALCULATION

C-3.1 Calculate the relative density as follows:

$$\text{Relative density, } t^{\circ}\text{C}/27^{\circ}\text{C} = \frac{m_2 - m}{(m_2 - m) - (m_3 - m)} \times d$$

where

t = temperature of the water in °C,

m = mass in g of empty stoppered pyknometer,

m_1 = mass in g of stoppered pyknometer filled with distilled water,

m_2 = mass in g of stoppered pyknometer and sample,

m_3 = mass in g of stoppered pyknometer with sample and distilled water, and

d = density of water at 27°C.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF LOSS ON IGNITION

D-1 PROCEDURE

Weigh accurately about 3 g of the material previously dried at 105 ± 2°C to constant mass in a previously ignited porcelain crucible of 50 ml capacity and ignite at a temperature of 850 ± 20°C. Cool to about 100°C and then finally transfer the crucible with its contents to a desiccator till the temperature comes down to room temperature, and weigh. Repeat the process until constant mass is obtained.

D-2 CALCULATION

$$\text{Loss on ignition at } 850^{\circ}\text{C} = \frac{100 \times (m_1 - m_2)}{m_1}$$

where

m_1 = mass, in g, of the sample taken for test, and

m_2 = mass, in g, of the sample after ignition.

ANNEX E

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF ZINC OXIDE

E-1 REAGENTS

E-1.1 Ammonium Fluoride

A.R. Grade.

E-1.2 Standard Zinc Solution

0.025 M.

Dissolve by heating in a beaker 1.635 g of pure zinc metal in a minimum hydrochloric acid (1:1). Transfer the solution to a one litre calibrated flask. Dilute and neutralize the solution with ammonia (1:1) in presence of methyl orange indicator. Acidify with hydrochloric acid (1:30) and add to the flask 20 ml of acetate buffer (*p*H 5.3). Finally dilute with water to one litre mark.

E-1.3 Buffer Solution

*p*H 5.3. Dissolve 21.5 g of sodium acetate with 2 ml of acetic acid in 300 ml of water and dilute to one litre.

E-1.4 Methyl Orange

0.05 percent (*m/v*) in Ethanol.

E-1.5 Xylenol Orange

0.1 percent (*m/v*) in water acidified with 2-3 drops of hydrochloric acid (1:3).

E-1.6 Standard EDTA Solution

Dissolve about 18.64 g of di-sodium salt of EDTA in water, transfer to a 1 000-ml calibrated flask and dilute to the mark.

E-2 PREPARATION OF STOCK SOLUTION

Weigh accurately about 0.5 g of the dried sample and transfer it to a beaker. Add 50 ml of water and 10 ml of hydrochloric acid (1:1). Wash on a hot plate and filter into a 250-ml calibrated flask. Wash the residue several times (5-6 times) with hot water. After cooling the solution in the calibrated flask, dilute to 250-ml mark.

E-3 PROCEDURE

Pipette an aliquot of 25 ml stock solution (**E-1**) into a 250-ml conical flask. Add an excess of standard EDTA solution (25 ml). Neutralize the solution with dilute ammonia (1:1) in presence of methyl orange and slightly acidify with dilute hydrochloric acid (1:3). Then add 10 ml to 15 ml of buffer (*p*H 5.3) and a few drops of xylenol orange indicator. At this stage, the solution should be yellow in colour. Then add one gram of ammonium fluoride, boil the solution for five minutes, cool to room temperature [if the solution develops some red colour add a few drops of dilute hydrochloric acid (1:3) for a colour change to yellow]. Titrate the solution against standard zinc solution. The end point is indicated by an abrupt change of colour from yellow to orange. Note the titre value (v_1). Also carry out a similar titration for the blank solution prepared in the same way as the test solution but without the sample and note the titre value (v_2).

E-4 CALCULATION

$$\text{Zinc oxide, percent by mass} = \frac{(v_2 - v_1) \times A \times 1\,000}{m} \times 1.444\,B$$

where

v_2 = volume in ml of zinc solution for titration of the blank;

v_1 = volume in ml of zinc solution for titration of the test solution;

A = equivalent ZnO in g/ml of standard zinc solution;

m = mass in g of the sample taken; and

B = percent Fe as determined in the sample (*see* Annex G).

NOTE — The above method is selective for determination of ZnO content present in commercial zinc oxide, because, it takes care of Al_2O_3, TiO_2, CaO, BaO and MgO which may contaminate during manufacture of zinc oxide.

ANNEX F

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF ACIDITY

F-1 REAGENTS

F-1.1 Standard Sodium Hydroxide Solution

0.01 N.

F-1.2 Phenolphthalein Indicator

Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit (*see* IS 323) and make up to 100 ml with water.

F-2 PROCEDURE

Weigh accurately about five grams of the material into a 400-ml beaker and boil with 200 ml of ammonia-free water for five minutes. Cool and make up to 250-ml in a volumetric flask. Filter through a double filter paper (Whatman No. 40 or its equivalent) and reject the first 100 ml of filtrate, collect the next 50 ml and titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

F-3 CALCULATION

Acidity (as H_2SO_4), percent by mass $= \frac{24.5\, v\, N}{m}$

where

v = volume in ml of standard sodium hydroxide solution;

N = normality of the standard sodium hydroxide slution; and

m = mass in g of the sample taken for the test.

ANNEX G

[*Table* 1, *Sl No.* (vii) and *Clause* E-4]

DETERMINATION OF IRON

G-1 APPARATUS

G-1.1 Nessler Cylinders

See IS 4161.

G-2 REAGENTS

G-2.1 Ammonium Thiocyanate Indicator Solution

2 percent (*m*/*v*).

G-2.2 Standard Iron Solution

Dissolve 1 g of pure iron in concentrated hydrochloric acid. Dilute the solution to about 100 ml. Transfer to a 1 000-ml volumetric flask, dilute to the mark and mix well. Dilute 100 ml of this solution to 1 000 ml in a volumetric flask. One millilitre of this dilute solution is equivalent to 0.1 mg of iron (as Fe). Alternately, dissolve 7.02 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4.6H_2O$] in water containing 10 ml of concentrated sulphuric acid and dilute ith water to 1 000 ml. One millilitre of this solution contains 1.0 mg of iron (as Fe).

G-3 PROCEDURE

G-3.1 Weigh accurately about 1.0 g of the material, mix it with 4 g of anhydrous sodium carbonate (free from iron) and fuse in a platinum crucible until the melt is clear. Pour the hot molten mass on a platinum dish and rotate so as to form a thin film. Place the crucible in the dish with the fused mass. Add about 10 ml of water and heat on a water-bath until the product is disintegrated. Remove the crucible and wash. Evaporate to dryness. Add a mixture of six millilitres of concentrated sulphuric acid and one millilitre of concentrated nitric acid. Evaporate the fumes of sulphur trioxide (SO_3). Dilute to about 50 ml and transfer into Nessler cylinder. Add 10 ml of thiocyanate indicator and dilute to 100 ml with water. Mix thoroughly. In the other Nessler cylinder add one millilitre of standard iron solution and carry through the entire analysis in the same manner as done with the material and make up the volume to 100-ml mark.

G-3.2 The material shall be regarded to have satisfied the requirement if the colour of the test solution is lighter than obtained in the standard solution.

G-3.3 Iron content in the sample can be further determined by matching the colour produced with the sample solution in the Nessler cylinder with that of known iron content.

ANNEX H

[*Table* 1, *Sl No.* (viii)]

TEST FOR THE PRESENCE OF METALLIC ZINC

H-1 APPARATUS

H-1.1 Glass Dish

Flat bottom, diameter approximately 6.25 cm.

H-1.2 Hot Water-Bath

H-2 REAGENTS

H-2.1 Anthraquinone

H-2.2 Sodium Hydroxide Solution

20 percent (*m/v*).

H-3 PROCEDURE

H-3.1 Mix 10 g of zinc oxide thoroughly with 0.1 g of anthraquinone and transfer to a glass dish. Add 20 ml of sodium hydroxide solution and stir the mass gently with a glass rod; on standing for five minutes blue spots appear. These spots appear both on the surface of the paste and underneath it. Place the dish in a bath of boiling water for two minutes. The spots become pinkish brown and sink though the thin layer of paste. Observe the spots on the bottom of the dish. Large particles may be seen on the bottom as well as on the top.

H-3.2 There shall not be more than 50 spots for 10 g of zinc oxide while hot and when examined visually.

ANNEX J

(Foreword)

COMMITTEE COMPOSITION

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Shri B. K. Biswas	National Test House, Calcutta
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Dr Devendra Mohan
Additional Director (Chem), BIS

(*Continued on page* 9)

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Test Affected

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